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A RAPID VAT-SIDE TEST FOR ASSAYING
TOXAPHENE IN CATTLE DIPS

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Toxaphene at a concentration of 0.5 percent in sprays and dips has proved satisfactory for the control of many external parasites affecting livestock, including ticks and certain flies (Bushland et al. 1, Division of Insects Affecting Man and Animals 2, Laake 4, Simon 5.

When toxaphene is to be used as a dip the concentration must be closely controlled; too much toxaphene may kill young animals and too little will not kill the ticks. The concentration may be altered by evaporation of water, leakage, dilution by rain or ground water, and sorption of the toxicant on the hair and skin of cattle passing through the bath. Consequently a method for ascertaining the strength of the bath is necessary. Such a test should be simple enough so that it can be successfully executed with a minimum of apparatus and in a very short time by an operator without any chemical knowledge.

The development of a vat-side test for toxaphene is handicapped by the lack of chemical methods for the determination of this insecticide. Of the physical methods that might be employed, measurement of the specific gravity of solutions of toxaphene in an organic solvent seemed to offer the most promise, and the present method is based upon this physical determination. A method of this kind has been proposed by Harris(3) for the determination of fats.

In a procedure of this type, using a solvent to extract the toxaphene from the dip and measuring the specific gravity of the extract, the greater the difference in specific gravity between the solvent and toxaphene, the more sensitive the test will be. Of the solvents investigated--n-hexane, n-heptane, isooctane (2, 2, 4-trimethylpentane), and benzene--isooctane was selected for use in the test because (1) it has a low specific gravity and will rise rapidly and separate easily from the aqueous phase, (2) it does not form emulsions readily, (3) it has a higher flash point and boiling point than the other solvents considered, and (4) it extracts the toxaphene from the dip giving solutions that differ appreciably in specific gravity from that of isooctane alone.

The dilute toxaphene emulsion used for dipping contains emulsifying agents which on shaking emulsify the solvent being used to extract the toxaphene; consequently, the dip emulsion must be broken before the extraction. Sodium chloride was selected from the various materials tested for this purpose, because it gave the most satisfactory results and is cheap and easily obtained.

Hydrometer readings taken over a temperature range from about 40° to 100° F. showed a straight-line relationship between temperature and specific gravity for isooctane and for isooctane solutions of toxaphene equivalent to extracts from 0.4- and 0.6-percent toxaphene dips based on an 8:1 ratio by volume of dip to solvent (fig. 1). The temperature-specific gravity lines are parallel; therefore, the difference in specific gravity between isooctane and the extracts is the same at any temperature within this range.

For ease in reading and convenience in size, an API hydrometer with a 45-90 scale has been chosen for the specific gravity readings. The values on this scale differ from specific gravity figures, a difference in reading of 7 corresponding to a 0.4-percent toxaphene dip and a difference of 10 to a 0.6-percent dip (fig. 2).

Differences in API hydrometer readings (fig. 2) compared with the percentages of toxaphene found in dip samples by analysis for organically bound chlorine for dips containing from about 0.1 to 1.0 percent of toxaphene, show the method to have an accuracy of ± 0.05 percent within this range. The slope of the line in figure 2 shows that the API difference may be converted to percentage of toxaphene by the factor 0.06.

A study of the influence of the oil used in the emulsion concentrate has shown that where the API gravity of the oil is about 45 or greater no correction is necessary.

METHOD OF ANALYSIS

Apparatus

- 1 1-liter graduate mixing cylinder. The glass stopper is replaced with a rubber or neoprene stopper.
- 1 hydrometer, API scale 45-90, 7-inch length. The hydrometer is enclosed in a testing tube of the syringe type, such as are used for testing automobile radiators or wet-cell batteries.
- 1 100-ml. graduated cylinder.

The primary objective of this investigation was to determine whether a method could be developed, and only slight consideration was given to the most practical field devices. The apparatus and procedure described here has been in use since May 18, 1949. Another type of apparatus, consisting of a conical plastic flask, a plastic tube threaded to fit the flask, and a threaded stopper to use in the flask for shaking and in the

tube for measuring, has been developed and used by a commercial laboratory since early in September 1949 for analyzing vat samples that contain their particular product. The hydrometer readings are taken by inserting the tube in the flask, adding water to float the isooctane in the narrow tube, and placing the hydrometer directly therein.

Reagents

Sodium chloride, ordinary table salt.

Isooctane (2, 2, 4-trimethylpentane), pure grade.

Procedure

Pour 800 ml. of the vat sample (kept well agitated during sampling) into a 1-liter graduate mixing cylinder. Add solid sodium chloride without agitation to bring the volume of the cylinder contents up to 850 ml. Stopper the cylinder and shake to dissolve the salt. Measure 100 ml. of isooctane in the 100-ml. graduated cylinder and pour into the mixing cylinder containing the sample. Replace the stopper in the mixing cylinder and shake vigorously for 3 minutes, after which set the cylinder upright and allow to stand for the separation of the isooctane layer. At the end of about 2 minutes swirl the top of the cylinder once or twice by keeping the bottom on the ground or bench and giving the top a slight rotary motion. The same effect may be obtained by tapping the upper part of the cylinder several times. Allow the cylinder to stand until enough of the isooctane extract has separated to make a specific gravity reading. Repeat the swirling action or tapping again in 3 or 4 minutes if necessary.

While waiting for the extract to separate, obtain the API gravity reading for isooctane by pouring a sufficient volume into the 100-ml. cylinder and drawing it up into the hydrometer syringe tube to float the hydrometer. Return the isooctane to the cylinder and keep for cleaning the syringe after reading the extract. Flush the syringe with air to remove adhering isooctane. When sufficient extract solution has separated, take a sample with the syringe and obtain the API gravity reading. Return the sample to the cylinder, flush the syringe with the isooctane from the 100-ml. cylinder, and discard the wash solution.

Determine the difference in the API readings between isooctane and the extract. A difference of 7 to 10 indicates a toxaphene content in the dip sample of 0.4 to 0.6 percent. As may be seen from figure 2, since the accuracy of the method is ± 0.05 percent, an API difference of 8.5 indicates 0.5 ± 0.05 percent of toxaphene.

FIELD USE OF TEST

Although the method was originally intended to be used for ascertaining whether a vat sample contained toxaphene within the limits of 0.4 to 0.6 percent, the standard curve (fig. 2) shows its reliability for concentrations up to 1 percent. Results obtained by H. V. Claborn in Texas on vat samples are shown in figure 3. The API differences are plotted against the percentages of toxaphene calculated from organically bound chlorine. The line drawn from the origin is in agreement with the standard curve (fig. 2). The dotted lines show the area of the graph lying within ± 0.05 percent of the standard curve. The several vats had been charged with five different emulsion-concentrate formulations; consequently, the results show that the test is not influenced by the oil used in the concentrate. Deviation from the curve may be accounted for by (1) incomplete extraction of the toxaphene by the isooctane, (2) poor sampling, or (3) reading of the API gravity before the isooctane layer is clear.

During the summer of 1949 samples were taken from vats in Florida. The results of the vat-side test in comparison with the concentration of the dip determined by analysis for organically bound chlorine are shown in table 1. In the main the agreement is within ± 0.05 percent. In the latter part of October and early in November 1949 vats were tested by the vat-side test in Florida and samples of dip sent to the laboratory, where the vat-side test was repeated and analysis made for organically bound chlorine in the isooctane extract and in the dip sample and the toxaphene content calculated. These results are shown in table 2. Agreement is seen between the vat-side tests made in the field and in the laboratory. All the vat-side tests reported in these tables were made in less than 15 minutes.

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SUMMARY

A simple vat-side test for assaying the strength of a stock dip containing toxaphene has been described. The percentage concentration of toxaphene in the dip is determined from the specific gravity of a 100-ml. isooctane extract of an 800-ml. vat sample. Differences in API gravity reading between the extract and isooctane of 7 to 10 indicate from 0.4 to 0.6 percent of toxaphene in the sample.

The apparatus and chemicals used are simple, inexpensive, and easily obtainable.

The test may be performed in a very short time, without regard to temperature, by a person who has no chemical training.

Although the test was devised to tell whether a dip contained 0.4 to 0.6 percent of toxaphene, it appears to be accurate to ± 0.05 percent in a toxaphene concentration up to 1.0 percent.

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Table 1. --Toxaphene content of dip samples from vats in Florida (summer 1949) as determined by analysis for organically bound chlorine and by vat-side test

Date (1949)	Number of cattle dipped	Sample	API reading difference in laboratory	Percentage of toxaphene in dip	
				From standard curve	By analysis <u>1/</u>
Vat A, Flagler County					
June 28(ca)	0	A-1-1	6.5, 7.0	0.39, 0.42	0.40
	400 (ca)	A-1-2	7.5, 7.0	.45, .42	.37
	600 (ca)	A-1-3	6.5, 6.5	.39, .39	.40
	800 (ca)	A-1-4	6.3, 6.0	.38, .36	.37
	1000 (ca)	A-1-5	6.0, 5.5	.36, .33	.33
July 26	0	A-2-1	7, 7	.42, .42	.32
	195	A-2-2	7-, 7	.41, .42	--
	388	A-2-3	7-, 7	.41, .42	--
	588	A-2-4	6.5, 7-	.39, .41	--
	719	A-2-5	6.5, 6.5	.39, .39	.42
Vat B, Volusia County					
July 21	0	B-1-1	6+, 6	.37, .36	.29
	400	B-1-3	7+, 6.5	.43, .39	.32
	489	B-1-4	7, 6	.42, .36	.37
	--	B-1-5	7+, 6	.43, .36	.37
Aug. 18(ca)	400	B-2-2	6, --	.36, --	.38 <u>2/</u>
	300	B-3-2	6.5, --	.39, --	.39 <u>2/</u>
	616	B-3-3	6.0, --	.36, --	.37 <u>2/</u>
Vat C, Orange County					
July 22	0	C-1-1	7.8, 7	.46, .42	.20
	200	C-1-2	8, 7	.48, .42	.25
	400	C-1-3	7, 6.5	.42, .39	.32
	600	C-1-4	6, --	.36, --	.36
	800	C-1-5	5.5, --	.33, --	.37
	1055	C-1-6	5+, 5	.31, .30	.37

^{1/} Analysis of dip by Carl Banks except as indicated.

^{2/} Analysis of isooctane extract by P. E. Hubanks.

Table 2. --Toxaphene content of dip samples from vats in Florida (October 1949) as determined by analysis for organically bound chlorine and by vat-side test

Sample	API reading difference		Percentage of toxaphene in dip		
	At vat-side	In laboratory	Vat-side test (± 0.05 percent)	From isooctane extract	From dip sample
T-1	6	6	0.36	0.38	0.40
T-2	7	7.5	.45	.43	.42
P-1	11	10	.60	.59	.60
P-2	10	8.5	.51	.51	.55
		8.5	.51	.51	.56
K-1	9.5	10	.60	.62	.55
		9.5	.57	.59	
K-2	10	10	.60	.61	.63
S-1	8	7+	.42	.44	
		7	.42	.38	.32
S-2	8	7	.42	.44	
		6.5	.39	.37	.29
Se-1	6	6.5	.39	.37	
		6	.36	.35	.42
H-3	6	7	.42	.43	
		7	.42	.43	.63
M-1	6	5	.30	.31	.36
W-1	9	9	.54	.53	.51
Se-2	8	7.5	.45	.44	
		7.5	.45	.43	.47
Se-3	7	7	.42	.41	
		7	.42	.41	.48
Se-4	6	6.5	.39	.41	.50
		7-	.42	.40	.50
MA-1	-	8-	.48	.47	
		7+	.42	.41	.47
MA-2	-	7.5	.45	.46	
		7.5	.45	.44	.48
MA-3	-	7	.42	.44	
		7	.42	.44	.43
MA-4	-	7-	.42	.43	
		7	.42	.43	.43

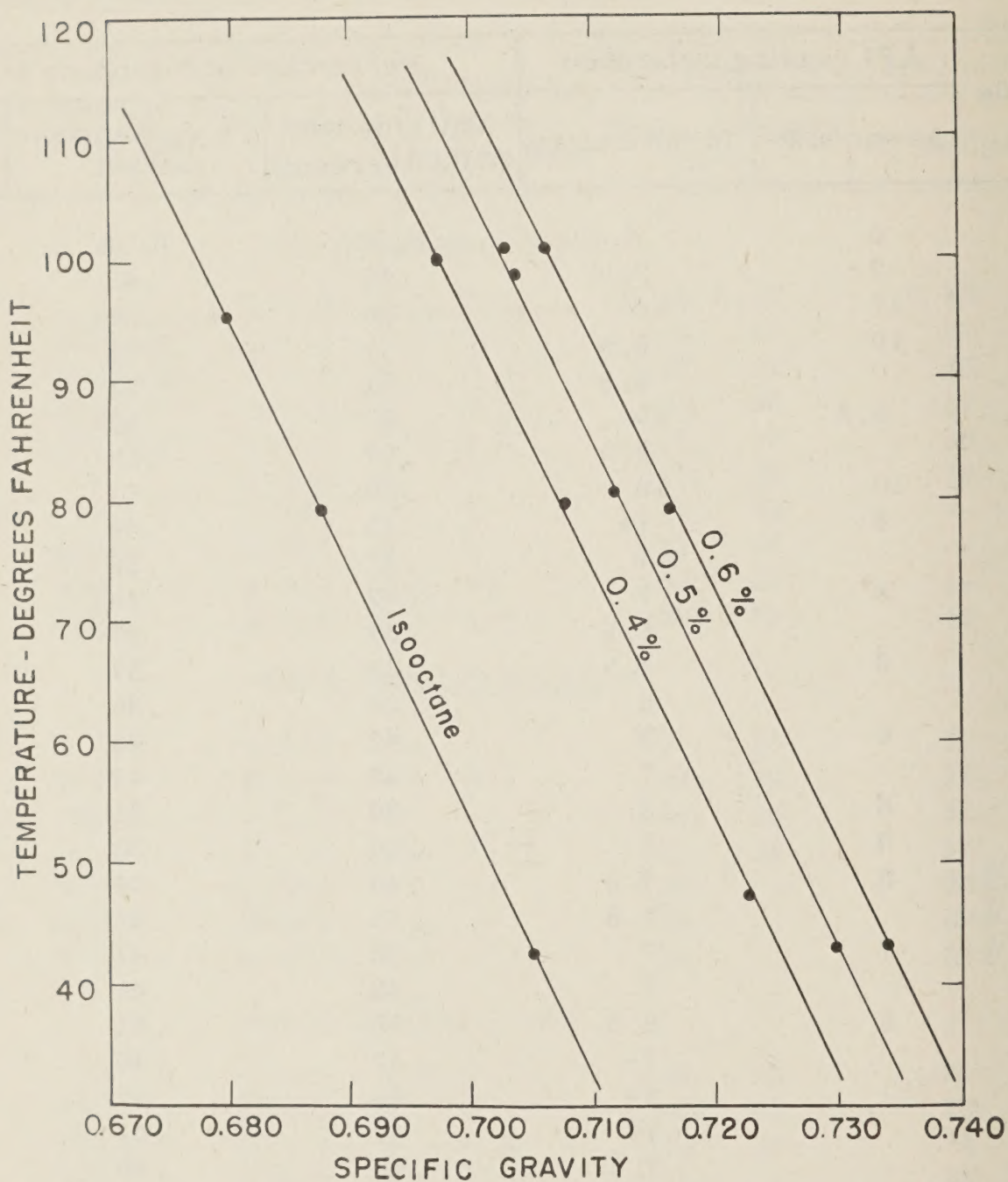


Figure 1. --Temperature-specific gravity curves. Toxaphene in isooctane. Percentage given is for dip. Ratio of sample to isooctane 8:1.

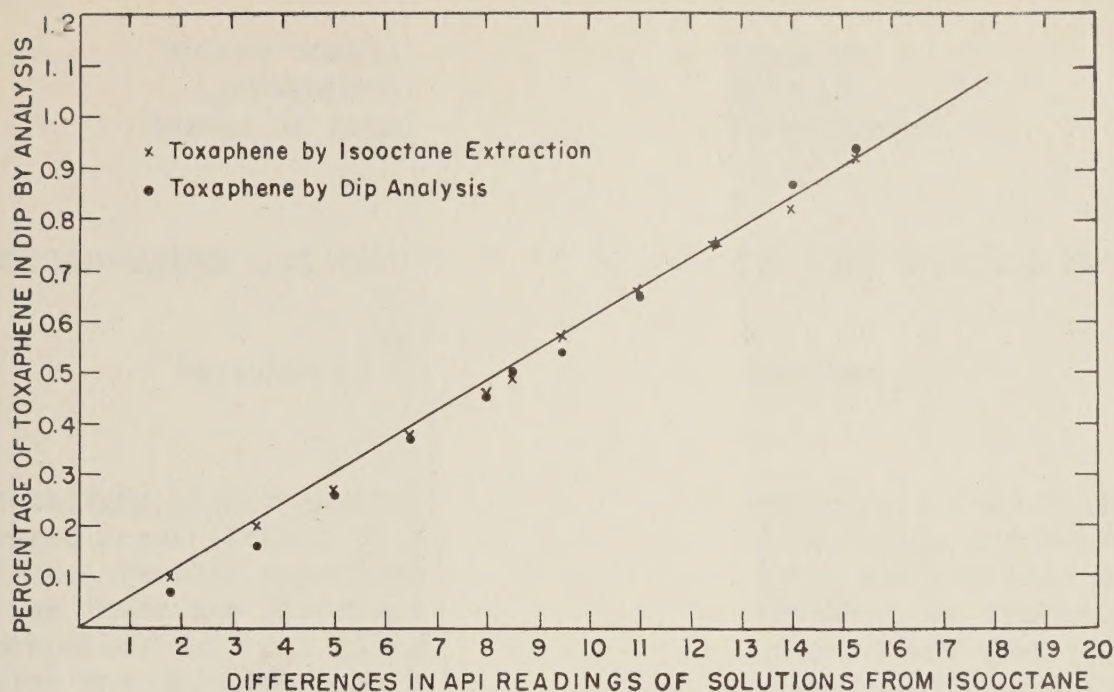


Figure 2. --Standard curve for toxaphene present in cattle dip. Emulsions by differences in API readings from isooctane. Ratio dip to solvent 8:1.

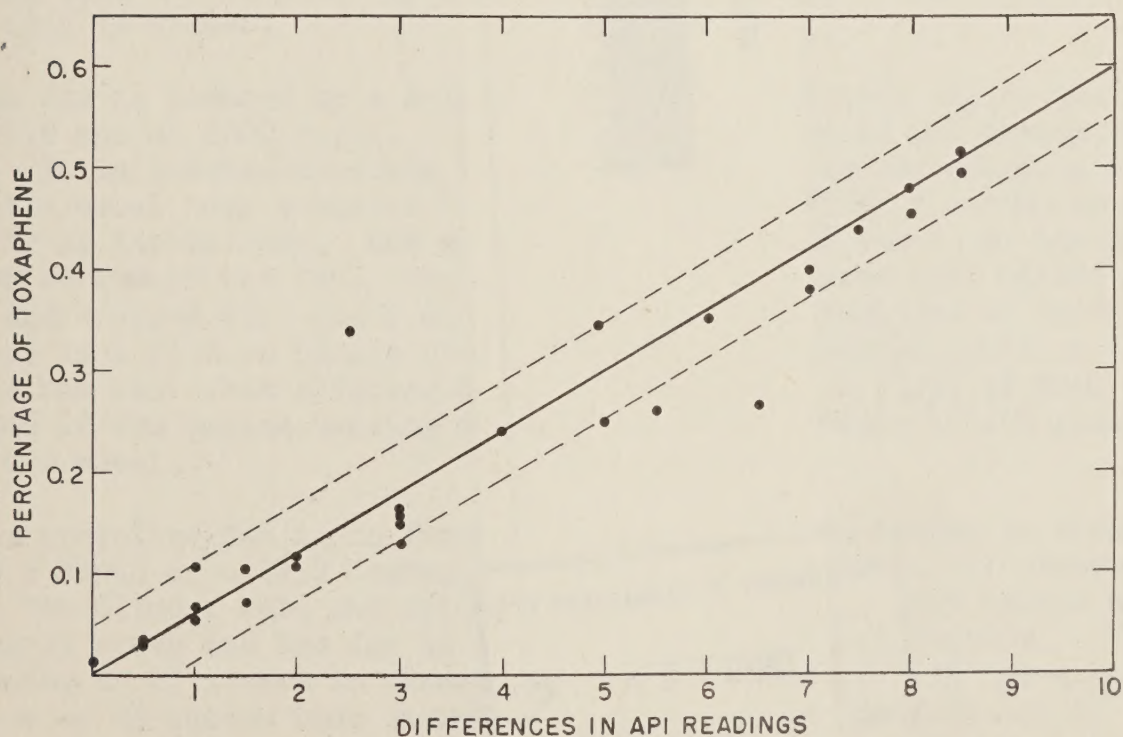


Figure 3. --Comparison of vat-side test with analysis of dip (Texas 1949).

